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Analytical Report

Fluorochemical Characterization of Aqueous Samples MPI Report No. L0019519

Testing Laboratory

MPI Research, Inc. 3058 Research Drive State College, PA 16801

Requester

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1 Introduction

Results are reported for the analysis of eight water samples received at MPI Research from the University of Georgia. The MPI Research study number assigned to the project is L0019519. Table I lists the target analyte quantitated for the sample.

Table I. Target Analyte for Quantitation

Compound Name	Acronym	Analysis Method
Perfluorobutyric Acid	C4 Acid	LC/MS/MS
Perfluoropentanoic Acid	C5 Acid	LC/MS/MS
Perfluorohexanoic Acid	C6 Acid	LC/MS/MS
Perfluoroheptanoic Acid	C7 Acid	LC/MS/MS
Perfluorooctanoic Acid	C8 Acid	LC/MS/MS
Perfluorononanoic Acid	C9 Acid	LC/MS/MS
Perfluorodecanoic Acid	C10 Acid	LC/MS/MS
Perfluoroundecanoic Acid	C11 Acid	LC/MS/MS
Perfluorododecanoic Acid	C12 Acid	LC/MS/MS
Perfluorotridecanoic Acid	C13 Acid	LC/MS/MS
Perfluorotetradecanoic Acid	C14 Acid	LC/MS/MS
Perfluorobutanesulfonate	C4 Sulfonate	LC/MS/MS
Perfluorohexanesulfonate	C6 Sulfonate	LC/MS/MS
Perfluoroheptanesulfonate	C7 Sulfonate	LC/MS/MS
Perfluorooctanesulfonate	C8 Sulfonate	LC/MS/MS
Perfluorodecanesulfonate	C10 Sulfonate	LC/MS/MS
Perfluorooctanesulfonamide	FOSA	LC/MS/MS
2-(N-methylperfluorooctanesulfonamido)	MeFOSAA	LC/MS/MS
2-(N-ethylperfluorooctanesulfonamide)	EtFOSAA	LC/MS/MS
N-methylperfluorooctane	MeFOSE	LC/MS/MS
N-ethylperfluorooctane	EtFOSE	LC/MS/MS
6:2 Fluorotelomer alcohol	6:2 FTOH	GC/MS
7:2 sFluorotelomer alcohol	7:2s FTOH	GC/MS
8:2 Fluorotelomer alcohol	8:2 FTOH	GC/MS
10:2 Fluorotelomer alcohol	10:2 FTOH	GC/MS

2 Sample Receipt

Eight water samples were received from the client cooled with wet ice on 11/16/09 and given the MPI Research login number of L0019519. The samples were stored refrigerated from receipt until analysis. Chain-of-custody information is presented in Attachment A.

3 Methods - Analytical and Preparatory

3.1 Water Sample Preparation for LC/MS/MS

Ten milliliters of sample was transferred into a 50 mL centrifuge tube. Ten milliliters of acetonitrile was added to the sample. After shaking, the sample was sonicated for approximately 2 hours then centrifuged at 3000 rpm for ~10 minutes. A 1 mL portion of the supernatant was transferred to an autosampler vial and fortified with an internal standard solution. The samples were then analyzed using electrospray LC/MS/MS.

3.2 Water Sample Preparation for GC/MS

One hundred milliliters of sample was transferred into a 250 mL polypropylene bottle. Forty milliliter of methyl tert-butyl ether (MTBE) was added to the bottle. The bottle was capped and then shaken for one hour on a reciprocation shaker. The content of the bottle was poured into a 250 mL separatory funnel. The bottle was rinsed with approximately 10 mL fresh MTBE and the rinsate was added to the separatory funnel. The aqueous phase in the funnel was discarded. The organic phase was collected in a 300 mL flask and dried with sodium sulfate. The dried organic phase was then quantitatively transferred into a 50 mL polypropylene centrifuge tube and concentrated to 1 mL using a nitrogen evaporator. The extracted was transferred into a 2 mL GC vial and 10 µL of internal standard was added. The sample was then analyzed by GC/MS.

3.3 Sample Analysis by LC/MS/MS

In High Pressure Liquid Chromatography (HPLC), an aliquot of extract is injected and passed through a liquid-phase chromatographic column. Based on the affinity of the analyte for the stationary phase in the column relative to the liquid mobile phase, the analyte is retained for a characteristic amount of time. Following HPLC separation, mass spectrometry provides a rapid and accurate means for analyzing a wide range of organic compounds. Molecules are ionized, fragmented, and detected. The ions characteristic of the compounds are observed and quantitated against calibration standards.

An HP1100 system interfaced to an Applied Biosystems API 5000 LC/MS/MS was used to analyze the sample extracts for quantitation. A gradient elution through a Phenomenex Luna 3μ C8(2) Mercury, 20 x 4.0 mm column was used for separation.

The following gradient was performed for C4-C14 acids, PFBS, PFHS, PFHPS, PFOS, PFDS, FOSA, and ¹³C PFOA (m+4):

Mobile Phase (A):	2mM Ammonium Acetate in Water	
Mobile Phase (B):	Methanol	
<u>Time</u>	<u>%A</u>	<u>%B</u>
0.0	90	10
0.5	90	10
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100

6.1 90 10 10.0 90 10

The following gradient was performed for MeFOSAA, EtFOSAA, MeFOSE and EtFOSE:

Mobile Phase (A):	2mM Ammonium Acetate in Water	
Mobile Phase (B):	Methanol	
<u>Time</u>	<u>%A</u>	<u>%В</u>
0.0	75	25
0.5	75	25
2.0	10	90
5.0	10	90
5.1	0	100
6.0	0	100
6.1	75	25
10.0	75	25

The following parameters were used for operation of the mass spectrometer:

Parameter	Setting
Ionization Mode	Electrospray
Polarity	Negative
Transitions Monitored	213→169 (C4 Acid)
, and an	263—219 (C5 Acid)
	313→269 (C6 Acid)
	363→319 (C7 Acid)
	413→369 (C8 Acid)
	463→419 (C9 Acid)
	513→469 (C10 Acid)
	563→519 (C11 Acid)
	613→569 (C12 Acid)
	663→619 (C13 Acid)
	713→669 (C14 Acid)
	299→80 (PFBS)
	399→80 (PFHS)
	449→99 (PFHpS)
	499→80 (PFOS)
	59999 (PFDS)
	498→78 (FOSA)
	217→172 (Internal Std. ¹³ C PFBA (m+4))
	415→370 (Internal Std. ¹³ C PFOA (m+2))
	515→470 (Internal Std. ¹³ C PFDA (m+2))
	503→80 (Internal Std. ¹³ C PFOS (m+4))
	417→372 (Surrogate ¹³ C PFOA (m+4))
	570→419 (MeFOSAA)
	584→419 (EtFOSAA)
	616→59 (MeFOSE)
	630→59 (EtFOSE)
Gas Temperature	400°C
	.00

3.4 Sample Analysis by GC/MS

The extracts were injected into a gas chromatograph (GC) equipped with a narrow bore capillary column and mass selective detector. The GC was temperature programmed to separate the analytes, and the analytes eluted from the column were introduced to the mass selective detector and identified by comparing retention times and abundances of characteristic masses to that of known standards. Sample concentration was calculated by comparing the response of the characteristic mass relative to that of the calibration curve.

The GC/MS system was operated using the following conditions:

Instrument	Hewlett-Packard model 6890 Series Gas
	Chromatograph/model 5973 Mass Selective
	Detector
Column	HP-1, 30 m x 0.25 mm ID, 1.00 μm df
Oven Temperature	Hold at 60°C for 4 min., ramp at 20°C/min. to
	140°C, ramp at 40°C to 240°C, hold for 5 minutes
Injector Temperature	200°C
Transfer Line Temperature	280°C
Carrier Gas	Helium
Column Flow	1.0 mL/min, Constant
Injection Mode	Pulsed Splitless, 30psi for 1.5 min.
Injection Liner	4 mm ID Single Gooseneck packed with glass
	wool
Injection Purge Delay	1.5 min.
Purge Flow to Split Vent	50 mL/min.
Injection Volume	2 µL
Electron Multiplier Voltage	From ATUNE + 306V
MS Acquisition Mode	SIM
lons Monitored	MFOET (Internal Standard): m/z 448, m/z 466
	8:1 FTOH (Surrogate): m/z 363, m/z 431
	6:2 FTOH: m/z 344, m/z 363
	7:2s FTOH: m/z 319, m/z 355
	8:2 FTOH: m/z 405, m/z 463
	10:2 FTOH: m/z 505, m/z 544
Dwell Time	50ms for each ion
MS Temperature .	Quad: 150 °C, Source: 230 °C

4 Analysis by LCMSMS

4.1 Calibration

A 9-point calibration curve was analyzed at the beginning of the analytical sequence for C4-C14 acids, PFBS, PFHS, PFHPS, PFOS, PFDS, FOSA, and ¹³C PFOA (m+4). A continuing calibration verification (CCV) standard (0.250 ng/mL) was used to verify the accuracy of the calibration curve for the duration of the analytical run. At the minimum every tenth sample was

a CCV, not including solvent blanks. The calibration curve and the last passing CCV (70-130%) will then bracket acceptable samples. The calibration points were prepared at 0.0125, 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) for LC/MS/MS analysis. The ratio of the analyte concentration to the IS concentration versus the ratio of the analyte instrument response (area) to the IS response (area) was plotted for each point. Using linear regression with 1/x weighting, the slope, y-intercept and coefficient of determination (r^2) were determined. A calibration curve is acceptable if $r^2 \ge 0.985$.

A 9-point calibration curve was analyzed at the beginning of the analytical sequence for MeFOSAA, EtFOSSA, MeFOSE and EtFOSE. A continuing calibration verification (CCV) standard (0.250 ng/mL) was used to verify the accuracy of the calibration curve for the duration of the analytical run. At the minimum every tenth sample was a CCV, not including solvent blanks. The calibration curve and the last passing CCV (70-130%) will then bracket acceptable samples. The calibration points were prepared at 0.0125, 0.025, 0.050, 0.100, 0.250, 0.500, 1.0, 2.5 and 5.0 ng/mL (ppb) for LC/MS/MS analysis. The instrument response versus the concentration was plotted for each point. Using quadratic regression with 1/x weighting, the X variable 1 (a), X variable 2 (b), intercept (c) and coefficient of determination (r) were determined. A calibration curve is acceptable if $r^2 \ge 0.985$.

For the results reported here, calibration criteria were met. The calibration curve is included in the raw data in Attachment C.

4.2 Surrogates and Internal Standards

¹³C labeled-perfluorooctanoic acid (¹³C PFOA (m+4)) was used as a surrogate for the water samples.

¹³C PFOA (m+4) recoveries can be found in Attachment B.

¹³C PFBA (m+4) was used as the internal standard for the water samples for C4 – C6 Acids.

¹³C PFOA (m+2) was used as the internal standard for the water samples for C7 – C9 Acids.

 13 C PFDA (m+2) was used as the internal standard for the water samples for C10 – C14 Acids.

¹³C PFOS (m+4) was used as the internal standard for the water samples for PFBS, PFHS, PFOS and FOSA

4.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries between 70-130% or the data is rejected. For the results reported here, the laboratory control spikes were within the acceptable range. Laboratory control spike recoveries are given in Attachment B.

4.4 Matrix Spikes

Two matrix spikes were prepared for the water samples, one for C4-C14 acids, PFBS, PFHS, PFHpS, PFOS, PFDS, FOSA, and ¹³C PFOA (m+4) analysis, and one for MeFOSAA, EtFOSSA, MeFOSE and EtFOSE analysis. They were prepared by adding a known



concentration of the target analyte to a separate sample. Matrix spikes are used to assess method accuracy in the matrix. The matrix spike should show recoveries between 70-130%. For the results reported here, the matrix spike was within the acceptable range with the exception of:

L19519-1 (1-1) Spk C at 1.0 ng/mL for C11 acid, C12 acid, and C14 acid, which gave high recoveries.

L19519-1 (1-1) Spk C at 0.1 ng/mL for MeFOSAA and EtFOSAA, which gave high recoveries.

Since the matrix spike recoveries were bias high and the samples were non-detected the data is considered acceptable.

Matrix spike recoveries are given in Attachment B.

4.5 Duplicate

Laboratory duplicates were not performed as part of this study.

5 Analysis by GC/MS

5.1 System Suitability and Calibration

Three system suitability standards were analyzed at the beginning of the analytical sequence. The %RSD of the peak area of each analyte should be ≤ 20 .

A 6-point calibration was analyzed. The calibration standard analyses were interspersed throughout the analytical sequence. The calibration points were prepared at 0.1, 0.2, 0.5, 1.0, 2.0 and 5.0 μ g/mL, which are equivalent to 1, 2, 5, 10, 20 and 50 μ g/L (ppb) in samples. A calibration curve is acceptable if $r^2 \geq 0.985$.

For the results reported here, system suitability and calibration criteria were met. The system suitability and calibration curve are included in the raw data in Attachment C.

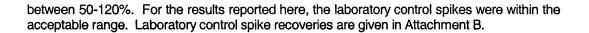
5.2 Surrogate and Internal Standard

1H,1H-Perfluoro-1-nonanol (8:1 FTOH) was used as surrogate standard. The recoveries of 8:1 FTOH can be found in Attachment B.

2-Perfluorooctyl-[1,1-2H2]-[1,2-13C2]-ethanol (MFOET) was used as internal standard.

5.3 Laboratory Control Spikes

Laboratory control spikes in the analytical set were prepared during each extraction set by adding a known concentration of the analyte to laboratory reagents. Laboratory control spikes are used to assess method accuracy. The laboratory control spikes must show recoveries





5.4 Matrix Spikes

Matrix Spike was prepared for sample 7-3 by adding a known concentration of the target analyte to a separate aliquot of sample. Matrix spike is used to assess method accuracy in the matrix. The matrix spike should show recoveries between 50-120%. For the results reported here, the matrix spike was within the acceptable range with the following exception:

The recovery of 10:2 FTOH was low and outside the acceptance limit, possibly due to sample matrix effect.

Matrix spike recoveries are given in Attachment B.

5.5 Duplicate

A laboratory duplicate sample was performed for sample 7-3.

6 Data Summary

6.1 GC/MS Sample Results

All of the surrogate recoveries were between the acceptance criteria of 50-120% except for sample 2-3. This sample had a surrogate recovery of 48%, which is marginally lower than the acceptance limit. The sample was not reanalyzed.

The results are reported in parts per billion (ng/mL) on an as-received basis.

Please see Attachment B for a detailed listing of the analytical results.

7 Data/Sample Retention

Samples are disposed of one month after the report is issued unless otherwise specified. All electronic data is archived on retrievable media and hard copy reports are stored in data folders maintained by MPI Research. Hardcopy data is stored for a minimum of five years. The client will be notified 30 days prior to the disposal of hardcopy data.



8 Attachments

- 7.1 Attachment A: Chain of Custody
- 7.2 Attachment B: Analytical Results
- 7.3 Attachment C: Raw Analytical Data (LC/MS/MS)
- 7.4 Attachment D: Raw Analytical Data (GC/MS)
- 9 Signatures

min. An	1-19-10
Mark Neeley, Research Chemist Associate II	Date
ashler E. Unasabu	1/19/2010
Ashley Unaegbu, Research Chemist Associate I	Date
Johns	1/18/2010
Xiang Zhu, Manager, Analytical	['] Date

Other Lab Member Contributed to the Report:

Robert Wolford



B







Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-1; 1-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	-ND	0.0125	12/11/2009
C5 Acid- Perfluoropentanoic Acid	ND	0.0125	12/11/2009
C6 Acid- Perfluorohexanoic Acid	0.0657	0.0125	12/11/2009
C7 Acid- Perfluoroheptanoic Acid	0.0301	0.0125	12/11/2009
C8 Acid- Perfluorooctanoic Acid	0.0776	0.0125	12/11/2009
C9 Acid- Perfluorononanoic Acid	NQ	0.0125	12/11/2009
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009
PFBS- Perfluorobutanesulfonate	0.0606	0.0125	12/12/2009
PFHS- Perfluorohexanesulfonate	0.0851	0.0125	12/12/2009
PFOS- Perfluorooctanesulfonate	0.520	0.0125	12/12/2009
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.0773	0.0125	12/11/2009
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	NQ	0.0125	12/11/2009

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).







Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-3; 1-3 Date of Extraction: 12/4/2009 Date Analyzed: 12/4/2009

ı	Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
	7-2s FTOH	ND	1.00
	6-2 FTOH	ND	1.00
	8-2 FTOH	ND	1.00
	10-2 FTOH	ND	1.00



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Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-4; 2-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	NQ	0.0125	12/11/2009
C5 Acid- Perfluoropentanoic Acid	NQ	0.0125	12/11/2009
C6 Acid- Perfluorohexanoic Acid	0.0659	0.0125	12/11/2009
C7 Acid- Perfluoroheptanoic Acid	NQ	0.0125	12/11/2009
C8 Acid- Perfluorooctanoic Acid	0.0720	0.0125	12/11/2009
C9 Acid- Perfluorononanoic Acid	0.0674	0.0125	12/11/2009
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009
PFBS- Perfluorobutanesulfonate	0.0825	0.0125	12/12/2009
PFHS- Perfluorohexanesulfonate	0.0269	0.0125	12/12/2009
PFOS- Perfluorooctanesulfonate	0.792	0.0125	12/12/2009
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	NQ	0.0125	12/11/2009
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.937	0.0125	12/11/2009
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	0.384	0.0125	12/11/2009

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).





Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-6; 2-3
Date of Extraction: 12/4/2009
Date Analyzed: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-7; 3-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date
C4 Acid- Perfluorobutyric Acid	ND	0.0125	12/11/2009
C5 Acid- Perfluoropentanoic Acid	ND	0.0125	12/11/2009
C6 Acid- Perfluorohexanoic Acid	NQ	0.0125	12/11/2009
C7 Acid- Perfluoroheptanoic Acid	NQ	0.0125	12/11/2009
C8 Acid- Perfluorooctanoic Acid	0.0709	0.0125	12/11/2009
C9 Acid- Perfluorononanoic Acid	ND	0.0125	12/11/2009
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009
PFBS- Perfluorobutanesulfonate	0.103	0.0125	12/12/2009
PFHS- Perfluorohexanesulfonate	NQ	0.0125	12/12/2009
PFOS- Perfluorooctanesulfonate	0.0747	0.0125	12/12/2009
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	0.0282	0.0125	12/11/2009
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.250	0.0125	12/11/2009
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	0.0846	0.0125	12/11/2009

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

 $[\]sqrt{Q}$ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-9; 3-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-10; 4-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date	
C4 Acid- Perfluorobutyric Acid	ND	0.0125		
C5 Acid- Perfluoropentanoic Acid	0.0384	0.0125	12/11/2009	
C6 Acid- Perfluorohexanoic Acid	0.0543	0.0125	12/11/2009	
C7 Acid- Perfluoroheptanoic Acid	0.0358	0.0125	12/11/2009	
C8 Acid- Perfluorooctanoic Acid	0.0763	0.0125	12/11/2009	
C9 Acid- Perfluorononanoic Acid	0.0403	0.0125	12/11/2009	
C10 Acid- Perfluorodecanoic Acid	NQ	0.0125	12/11/2009	
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009	
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009	
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009	
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009	
PFBS- Perfluorobutanesulfonate	0.0388	0.0125	12/12/2009	
PFHS- Perfluorohexanesulfonate	ND	0.0125	12/12/2009	
PFOS- Perfluorooctanesulfonate	2.00	0.0125	12/12/2009	
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009	
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009	
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009	
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	NQ	0.0125	12/11/2009	
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.0639	0.0125	12/11/2009	
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	0.121	0.0125	12/11/2009	

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-12; 4-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	1.10	1.00
8-2 FTOH	, ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-13; 5-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date	
C4 Acid- Perfluorobutyric Acid	NQ	0.0125		
C5 Acid- Perfluoropentanoic Acid	0.0331	0.0125	12/11/2009	
C6 Acid- Perfluorohexanoic Acid	0.0622	0.0125	12/11/2009	
C7 Acid- Perfluoroheptanoic Acid	0.0634	0.0125	12/11/2009	
C8 Acid- Perfluorooctanoic Acid	0.133	0.0125	12/11/2009	
C9 Acid- Perfluorononanoic Acid	0.0543	0.0125	12/11/2009	
C10 Acid- Perfluorodecanoic Acid	NQ	0.0125	12/11/2009	
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009	
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009	
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009	
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009	
PFBS- Perfluorobutanesulfonate	0.0897	0.0125	12/12/2009	
PFHS- Perfluorohexanesulfonate	ND	0.0125	12/12/2009	
PFOS- Perfluorooctanesulfonate	1.74	0.0125	12/12/2009	
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009	
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009	
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009	
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	0.0420	0.0125	12/11/2009	
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.554	0.0125	12/11/2009	
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	0.228	0.0125	12/11/2009	

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

 $[\]sqrt{Q}$ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).





Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-15; 5-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-16; 6-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date	
C4 Acid- Perfluorobutyric Acid	ND	0.0125		
C5 Acid- Perfluoropentanoic Acid	0.0536	0.0125	12/11/2009	
C6 Acid- Perfluorohexanoic Acid	0.0768	0.0125	12/11/2009	
C7 Acid- Perfluoroheptanoic Acid	0.0485	0.0125	12/11/2009	
C8 Acid- Perfluorooctanoic Acid	0.169	0.0125	12/11/2009	
C9 Acid- Perfluorononanoic Acid	0.106	0.0125	12/11/2009	
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009	
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009	
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009	
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009	
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009	
PFBS- Perfluorobutanesulfonate	0.0267	0.0125	12/12/2009	
PFHS- Perfluorohexanesulfonate	NQ	0.0125	12/12/2009	
PFOS- Perfluorooctanesulfonate	0.686	0.0125	12/12/2009	
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009	
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009	
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009	
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	NQ	0.0125	12/11/2009	
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	NQ	0.0125	12/11/2009	
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	12/11/2009	

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

 $[\]sqrt{Q}$ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).





Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-18; 6-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	3.30	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-19; 7-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date	
C4 Acid- Perfluorobutyric Acid	NQ	0.0125	12/11/2009	
C5 Acid- Perfluoropentanoic Acid	NQ	0.0125	12/11/2009	
C6 Acid- Perfluorohexanoic Acid	0.0496	0.0125	12/11/2009	
C7 Acid- Perfluoroheptanoic Acid	0.0309	0.0125	12/11/2009	
C8 Acid- Perfluorooctanoic Acid	0.0717	0.0125	12/11/2009	
C9 Acid- Perfluorononanoic Acid	0.0332	0.0125	12/11/2009	
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009	
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009	
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009	
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009	
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009	
PFBS- Perfluorobutanesulfonate	0.0285	0.0125	12/12/2009	
PFHS- Perfluorohexanesulfonate	ND	0.0125	12/12/2009	
PFOS- Perfluorooctanesulfonate	1.69	0.0125	12/12/2009	
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009	
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009	
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009	
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	NQ	0.0125	12/11/2009	
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	0.0619	0.0125	12/11/2009	
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	0.111	0.0125	12/11/2009	

ND = Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).



Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-21; 7-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	1.30	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-21 Dup; 7-3

Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	1.60	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorochemical Residues in Water Samples By LC/MS/MS

Sample ID: L0019519-22; 8-1 Date of Extraction: 12/11/2009

Analyte	Amount Found (ng/mL)	LOD (ng/mL)	Analysis Date	
C4 Acid- Perfluorobutyric Acid	ND	0.0125	12/11/2009	
C5 Acid- Perfluoropentanoic Acid	ND	0.0125	12/11/2009	
C6 Acid- Perfluorohexanoic Acid	ND	0.0125	12/11/2009	
C7 Acid- Perfluoroheptanoic Acid	ND	0.0125	12/11/2009	
C8 Acid- Perfluorooctanoic Acid	ND	0.0125	12/11/2009	
C9 Acid- Perfluorononanoic Acid	ND	0.0125	12/11/2009	
C10 Acid- Perfluorodecanoic Acid	ND	0.0125	12/11/2009	
C11 Acid- Perfluoroundecanoic Acid	ND	0.0125	12/11/2009	
C12 Acid- Perfluorododecanoic Acid	ND	0.0125	12/11/2009	
C13 Acid- Perfluorotridecanoic Acid	ND	0.0125	12/11/2009	
C14 Acid- Perfluorotetradecanoic Acid	ND	0.0125	12/11/2009	
PFBS- Perfluorobutanesulfonate	ND	0.0125	12/12/2009	
PFHS- Perfluorohexanesulfonate	ND	0.0125	12/12/2009	
PFOS- Perfluorooctanesulfonate	ND	0.0125	12/12/2009	
FOSA- Perfluorooctane sulfonamide	ND	0.0125	12/12/2009	
PFHpS- Perfluoroheptanesulfonate	ND	0.0125	12/12/2009	
PFDS- Perfluorodecanesulfonate	ND	0.0125	12/12/2009	
MeFOSAA- 2(N-Methylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
EtFOSAA- 2(N-Ethylperfluorooctanesulfonamido) acetic acid	ND	0.0125	12/11/2009	
MeFOSE- N-Methylperfluorooctane sulfonamidoethanol	ND	0.0125	12/11/2009	
EtFOSE- N-Ethylperfluorooctane sulfonamidoethanol	ND	0.0125	12/11/2009	

ID Not detected = Response is below the LOD of 0.0125 ng/mL (ppb).

IQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL (ppb).





Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-23; 8-3 Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Recovery Summary of Fluorochemical Residues in Water Samples

			C4 Acid			C5 Acid			C8 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.0915	92	ND	0.0897	90	ND	0.107	107
Reagent Spike B 1.0 ng/mL	1.00	ND	1.03	103	ND	1.02	102	ND	1.14	114
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	ND	0.817	82	ND	1.16	116	0.0657	1.30	123

			C7 Acid			C8 Acid			C9 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.108	108	ND	0.100	100	ND	0.110	110
Reagent Spike B 1.0 ng/mL	1.00	ND	1.13	113	ND	1.09	109	ND	1.11	111
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	0.0301	1.11	108	0.0776	1.19	111	NQ	1.12	112

			C10 Acid			C11 Acid			C12 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mt.	0.100	ND	0.101	101	ND	0.122	122	NĐ	0.112	112
Reagent Spike B 1.0 ng/mL	1.00	ND	1.06	106	ND	1.28	128	ND	1.22	122
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	ND	1.14	114	ND	1.49	149^	ND	1.43	143^

			C13 Acid			C14 Acid	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.113	113	ND	0.106	106
Reagent Spike B 1.0 ng/mL	1.00	ND	1.17	117	ND	1.21	121
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	ND	1.28	128	ND	1.54	154^

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.



NQ * Not quantifiable * Response is between the LOD and the LOQ of 0.0250 ng/mL.

[^] Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130%. The recovery is bias high and the result is non detect, therefore, the data is considered acceptable.





Recovery Summary of Fluorochemical Residues in Water Samples

			PFB\$			PFHS			PFOS	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.105	105	ND	0.0966	97	ND	0.0976	98
Reagent Spike B 1.0 ng/mL	1.00	ND	0.985	99	ND	1.05	105	ND	1.06	106
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	0.0606	1.08	102	0.0851	1.14	105	0.520	1.53	101

			FOSA			PFHpS			PFDS	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.102	102	ND	0.103	103	ND	0.0979	98
Reagent Spike B 1.0 ng/mL	1.00	ND	1.05	105	ND	1.06	106	ND	1.06	106
1-1 Spike C (L19519-1 Spk C, 1.0 ng/mL Lab Spike)	1.00	ND	0.901	90	ND	0.961	96	ND	1.08	108

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.
NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.





Recovery Summary of Fluorochemical Residues in Water Samples

			MeFOSAA			EtFOSAA	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found In Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.119	119	ND	0.118	118
Reagent Spike B 1.0 ng/mL	1.00	ND	1.04	104	ND	1.03	103
1-1 Spike C (L19519-1 Spk C, 0.1 ng/mL Lab Spike)	0.100	ND	0.140	140^	ND	0.190	190^

			MeFOSE			EtFOSE	
Sample Description	Amount Spiked (ng/mL)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)	Amt Found in Sample (ng/mL)	Amount Recovered (ng/mL)	Recovery (%)
Reagent Spike A 0.1 ng/mL	0.100	ND	0.102	102	ND	0.106	106
Reagent Spike B 1.0 ng/mL	1.00	ND	1.01	101	ND	1.04	104
1-1 Spike C (L19519-1 Spk C, 0.1 ng/mL Lab Spike)	0.100	0.0773	0.187	110	NQ	0.0995	100

ND = Not detected = Response is below the LOD of 0.0125 ng/mL.



NQ = Not quantifiable = Response is between the LOD and the LOQ of 0.0250 ng/mL.

[^] Laboratory Matrix Spike recovery is outside the acceptance criteria of 70 - 130%. The recovery is bias high and the result is non detect, therefore, the data is considered acceptable.





Recovery Summary of ¹³C PFOA (m+4) in Water Samples

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL, ppb)	Amount Recovered (ng/mL, ppb)	Recovery (%)
N/A	Reagent Control	1.00	1.03	103
N/A	Reagent Spike A	0.100	0.105	105
N/A	Reagent Spike B	1.00	1.06	106
1-1 Spike C	L19519-1 Spk C	1.00	1.05	105
1-1	L19519-1	1.00	1.00	100
2-1	L19519-4	1.00	1.12	112
3-1	L19519-7	1.00	1.07	107
4-1	L19519-10	1.00	1.09	109
5-1	L19519-13	1.00	1.11	111
6-1	L19519-16	1.00	0.959	96
7-1	L19519-19	1.00	0.904	90
8-1	L19519-22	1.00	1.08	108





Fluorotelomer Analysis by GC/MS

Sample ID: Method Blank
Date of Extraction: 12/4/2009

Analyte	Amount Found (ng/mL)	LOQ (ng/mL)
7-2s FTOH	ND	1.00
6-2 FTOH	ND	1.00
8-2 FTOH	ND	1.00
10-2 FTOH	ND	1.00





Fluorotelomer Analysis by GC/MS

Sample ID: Lab Control Spike Date of Extraction: 12/4/2009 Date Analyzed: 12/4/09

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery
7-2s FTOH	3.50	5.00	1.00	70
6-2 FTOH	7.50	9.88	1.00	76
8-2 FTOH	3.60	5.11	1.00	71
10-2 FTOH	3.80	5.03	1.00	76





Fluorotelomer Analysis by GC/MS

Sample ID: Lab Control Spike Duplicate

Date of Extraction: 12/4/2009 Date Analyzed: 12/4/09

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery	% RPD
7-2s FTOH	3.50	5.00	1.00	70	0.0
6-2 FTOH	7.70	9.88	1.00	78	2.6
8-2 FTOH	3.90	5.11	1.00	76	8.0
10-2 FTOH	3.90	5.03	1.00	78	2.6



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Fluorotelomer Analysis by GC/MS

Sample ID: L0019519-21 Matrix Spike; 7-3

Date of Extraction: 12/4/2009 Date Analyzed: 12/5/09

Analyte	Amount Found (ng/mL)	Amount Spiked (ng/mL)	LOQ (ng/mL)	% Recovery
7-2s FTOH	2.80	5.00	1.00	56
6-2 FTOH	10.00	9.88	1.00	101
8-2 FTOH	3.10	5.11	1.00	61
10-2 FTOH	1.70	5.03	1.00	34





Recovery Summary of 8:1 FTOH in Water Samples

Client Sample ID	MPI Sample ID	Amount Spiked (ng/mL, ppb)	Amount Recovered (ng/mL, ppb)	Recovery (%)
N/A	Method Blank	5.00	3.90	78
N/A	LCS	5.00	3.40	68
N/A	LCSD	5.00	3.20	64
1-3	L19519-3	5.00	3.60	72
2-3	L19519-6	5.00	2.40	48
3-3	L19519-9	5.00	2.80	56
4-3	L19519-12	5.00	2.50	50
5-3	L19519-15	5.00	2.80	56
6-3	L19519-18	5.00	2.70	54
7-3	L19519-21	5. 00	2.70	54
7-3 Duplicate	L19519-21 Duplicate	5.00	2.80	56
7-3 Spike	L19519-21 Matrix Spike	5.00	2.80	56
8-3	L19519-23	5.00	3.20	64